

SYNTHESIS, SORPTION PROPERTIES, AND EVALUATION OF SILICA GEL WITH ADSORBED POLY[8-OXYQUINOLINE METHACRYLATE] AS A SORBENT FOR Cu(II), Cd(II), Pb(II) AND Fe(III) IONS

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Abstract. In the present study, a novel functionalized silica composite was prepared by adsorption of poly[8-oxyquinoline methacrylate] on the surface of silica gel. Immobilization of poly[8-oxyquinoline methacrylate] on the silica gel surface was confirmed using IR spectroscopy and thermogravimetric analysis combined with mass spectrometry. Scanning electron microscopy revealed that the polymer almost completely covers the surface of the silica gel in the form of agglomerates of different geometric shapes. The processes of sorption of Cu(II), Cd(II), Pb(II), and Fe(III) ions on the surface of the synthesized composite in the static mode have been studied.

Keywords: adsorption, composite, silica gel, poly[8-oxyquinoline methacrylate], Cu(II), Cd(II), Pb(II), Fe(III) ions.

1. Introduction

Recently, adsorption has been widely utilized to remove heavy metals from aqueous solutions due to its many advantages, such as low cost, easy handling, high removal efficiency, and environmental friendliness.¹⁻³ There are a lot of materials, including polymer-modified silica gel,⁴⁻⁷ carbon nanocomposites,⁸ magnetic nanoparticles (Fe₃O₄),⁹ fibers,¹⁰ and modified natural clays^{11,12} that have been used as adsorbents to remove heavy metals from aqueous solutions.

Fixation of functional polymers on the surface of matrices of different chemical natures allows strengthening or changing their useful (electrical, catalytic, or sorption) properties. In particular, the immobilization of

polymers with nitrogen, sulfur, or oxygen-containing groups in the main chain, which can be used as ligands, allows obtaining adsorbents with complexing properties, which effectively remove cations of transition toxic metals from wastewater. Polydisperse synthetic inorganic matrices, which are widely used for this purpose are silica,^{13,14} carbonaceous materials (activated carbon, fullerenes, fluffy graphite), oxides of elements of group IV (GeO₂, SnO₂, TiO₂).

8-Hydroxyquinoline is one of the most famous chelating agents and its metal complexes have been known since the XIX century. Modification of the surface of porous inorganic matrices of different chemical nature with 8-oxyquinoline and its derivatives allows obtaining sorbents for ions of many transition metals, which are widely used in chromatography, hybrid and combined methods of analysis, etc.¹⁵⁻²¹ Polymers with oxyquinoline groups also show complex-forming activity with transition metal ions due to their coordination to nitrogen and quinoline oxygen atoms.²²

We performed *in situ* immobilization of poly[8-oxyquinoline methacrylate] on the surface of silica gel²³ and found that the sorption capacity of silica gel after *in situ* modification of poly[8-oxyquinoline methacrylate] increases relative to Cu (II), Cd (II), Fe (III) and Mn (II) ions.

This work is devoted to the synthesis and study of the sorption properties of silica gel with adsorbed poly[8-oxyquinoline methacrylate] on Cu (II), Cd (II), Pb (II), and Fe (III) ions and the comparison of the sorption properties of silica gels with *in situ* modified and adsorbed poly[8-oxyquinoline methacrylate] relative to these metals.

2. Experimental

2.1. Materials

Silica gel (fraction of 0.1–0.2 mm diameter particles, specific surface 428 m²/g, Merck) was used as the inorganic carrier of the composite. The synthesis of 8-oxyquinoline methacrylate was performed similarly.²³

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The synthesis of poly[8-oxyquinoline methacrylate] was carried out by initiating radical polymerization using as initiator 2,2'-azobisisobutyronitrile (AIBN) according to the following procedure:

6.5 g of recrystallized 8-oxyquinoline methacrylate was added to a 250 mL flat-bottomed flask, dissolved in 50 mL of DMF and 0.065 g of 2,2'-azobisisobutyronitrile was added, heated under vigorous stirring for 5 hours at 85–90 °C passing argon through the mixture. The mixture was left for a day, then precipitated in propanol-2. A white precipitate was obtained, which was dried at room temperature to a constant weight.

The synthesis of organo-mineral composites was carried out by adsorption of poly[8-oxyquinoline methacrylate] on the surface of silica gel according to the following method:

3.65 g of polymer was added to a 250 mL flat-bottomed flask and dissolved in 35 mL of tetrahydrofuran (THF). Then 10.95 g of silica gel was added to the resulting solution; silica gel was previously heated to a temperature of 60 °C under vigorous stirring on a magnetic stirrer for 5 hours, gradually passing argon through the mixture. A day later, the synthesized composite was decanted on a filter and dried at room temperature for several days to a constant weight.

2.2. Methods

FTIR spectra of the composite and the original silica gel were recorded using an IR spectrometer with Fourier transformation (Thermo Nicolet Nexus FT-IR, USA). For this purpose, the samples were ground in an agate mortar and pressed with KBr. The FTIR spectra were recorded in the spectral range of 500–4000 cm⁻¹ with 16 scans per spectrum at a resolution of 4 cm⁻¹.

Thermal analysis. The amount of immobilized polymer on the surface of the silica gel was evaluated by thermogravimetric analysis, which was obtained on a synchronous TG / DTA analyzer “Shimadzu DTG-60 H” (Shimadzu, Japan) in the temperature range 15–1000 °C. The heating rate of the samples was 10 deg/min.

Differential scanning calorimetry was performed on an instrument “STA 449 Jupiter F1” (Netzsch, Germany) with a mass spectroscopic attachment “QMS 403C” (Germany).

Low-temperature adsorption-desorption of nitrogen. The values of the specific surface area and the average pore diameter were calculated from the isotherms of low-temperature adsorption-desorption of nitrogen using the sorbometer software “ASAP 2420 V1.01” (Micromeritics, USA). Before measurement, the samples were degassed at 60 °C for 24 hours.

Surface morphology analysis. The surface morphology of the composite was observed by using a scanning

electron microscope (SEM, LEO 1430VP, Carl Zeiss, Germany).

2.2.1. Description of adsorption processes

Properties of the obtained composite to adsorb Cu(II), Pb(II), Cd(II), and Fe(III) were studied in a static mode. Working solutions of the nitrates of corresponding metals were prepared in volumetric flasks of 25 mL, 50 mL, or 100 mL, diluting the solutions to mark with standard buffer solutions of certain pH values. Then the required volume was added to flat-bottom flasks containing 0.1 g of the adsorbent. The reaction was proceeding while the flasks were shaken mechanically. Equilibrium concentrations of ions were measured using atomic absorption methods.

Working nitrate solutions of Cu(II), Pb(II), Cd(II), and Fe(III) were prepared with sets of “standard sample solutions” of those salts on 1M HNO₃ background (produced by A.V. Bogatsky FHI in Odesa) with concentrations of 1 and 10 mg/mL.

2.2.2. Analysis of ion concentrations

Sorption capacity (*A*) was calculated by the following equation:

$$A = (c_o - [M]) V / m \quad (1)$$

where *c_o* is the concentration of the metal in the starting solution, [*M*] is the concentration of the metal at equilibrium, *V* is the volume of the starting solution, *m* is the mass of the composite.

Atomic absorption spectrophotometer “C115-M” (Ukraine) in the flame of a mixture of “acetylene-air” was used to measure the equilibrium concentrations of copper (II), cadmium (II), lead (II), and iron (III). Equilibrium concentrations in the solutions were calculated by comparing the intensities of the spectral lines with those of standard solutions. Maxima wavelengths were: 324.7 nm for Cu(II), 228.8 nm – for Cd(II), 283.3 nm – for Pb(II), and 248.3 nm – for Fe(III), the aperture being 0.5 cm wide.

3. Results and Discussion

To confirm the immobilization of poly[8-oxyquinoline methacrylate] on the surface of silica gel, the method of IR spectroscopy was used. The IR spectra of the original and polymer-modified silica gels are shown in Fig. 1.

As a result of the analysis of the IR spectra of the synthesized composite and the original silica gel, new absorption bands can be seen in the spectrum of the synthesized composite in comparison with the spectrum of the original silica gel. Regarding the confirmation of the

presence of this polymer in the surface layer of silica gel, the most informative are a number of bands in the range of 1200-1750 cm^{-1} , which can be interpreted as follows:

- the absorption band at 1750 cm^{-1} corresponds to the valence vibrations of C = O group;
- absorption bands at 1050, 1070, 1250, and 1270 cm^{-1} correspond to the vibrations = C-O-C groups;
- the absorption band at 1698 cm^{-1} can be attributed to the valence vibrations ν (C-N) of the aromatic system of quinoline;
- absorption bands at 1498 cm^{-1} and 1418 cm^{-1} correspond to skeletal vibrations of C-C bonds of the aromatic system of quinoline;
- the absorption bands at 2840-3000 cm^{-1} correspond to the valence vibrations of the C-H bonds.

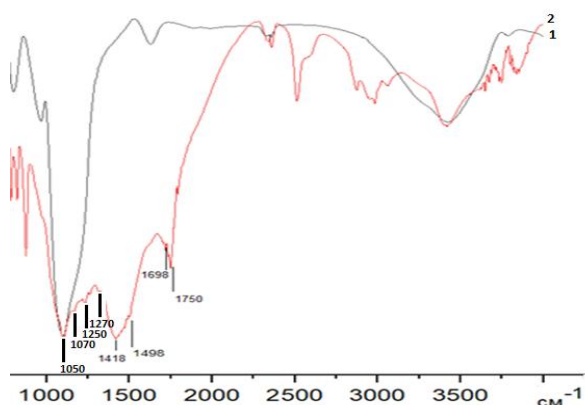


Fig. 1. IR spectra of the original silica gel (1) and the synthesized composite (2)

The presence of the corresponding bands in the IR spectrum of the synthesized composite may be evidence of the presence of poly[8-oxyquinoline methacrylate] in its composition.

To study the thermal stability of the adsorbed poly[8-oxyquinoline methacrylate] on the surface of silica gel and to determine its mass fraction in the composite, its thermogravimetric analysis was performed. The obtained thermogram is shown in Fig. 2. As can be seen from this figure, the thermal destruction of the adsorbed polymer occurs in the temperature range from 150 to 792 $^{\circ}\text{C}$ in 3 stages:

1) the decomposition of the polymer begins at a temperature of about 150 $^{\circ}\text{C}$;

2) in the temperature range from 356 to 792 $^{\circ}\text{C}$ is its partial oxidation with weight loss, accompanied by an endothermic effect;

3) the final thermo destruction of poly[8-oxyquinoline methacrylate] with a large exoeffect is observed at 792 $^{\circ}\text{C}$.

As a result of these processes, about 48.9 % of the composite mass is lost. As shown in our previous work,²³ the mass lost by the original silica gel in this temperature range is 5.4 %. Therefore, the mass of the adsorbed polymer is 43.5 %.

To detail the process of thermodestruction of the poly[8-oxyquinoline methacrylate] adsorbed on the surface of silica gel, its thermogram, which is combined with the mass spectrum, was obtained in 3D format (Fig. 3a). Several mass spectra at different temperatures were also obtained, one of which (at a temperature of 792 $^{\circ}\text{C}$) is shown in Fig. 3b.

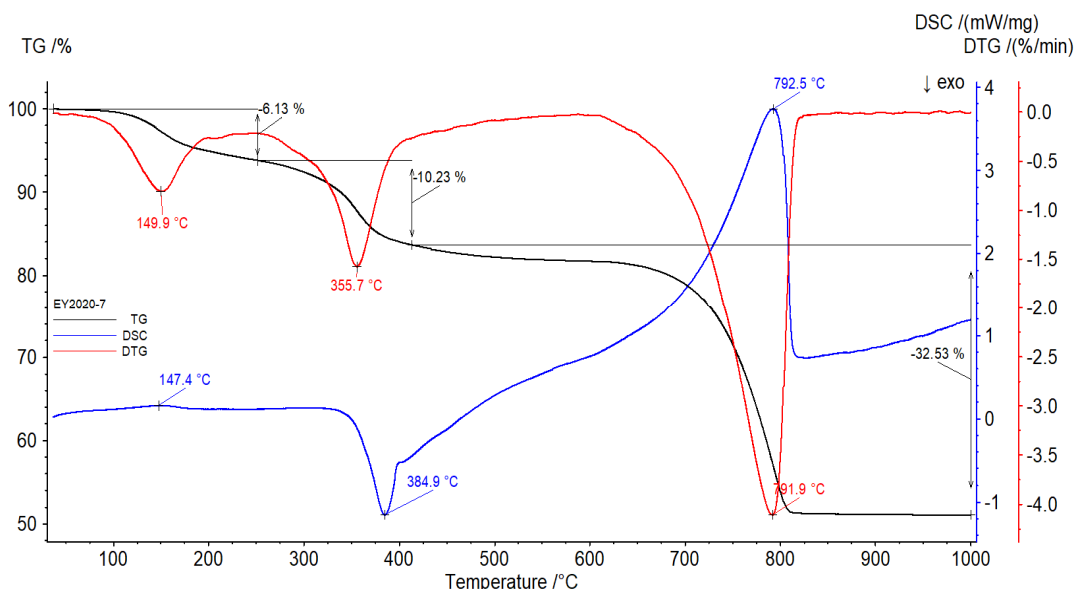


Fig. 2. Thermogram of the synthesized composite

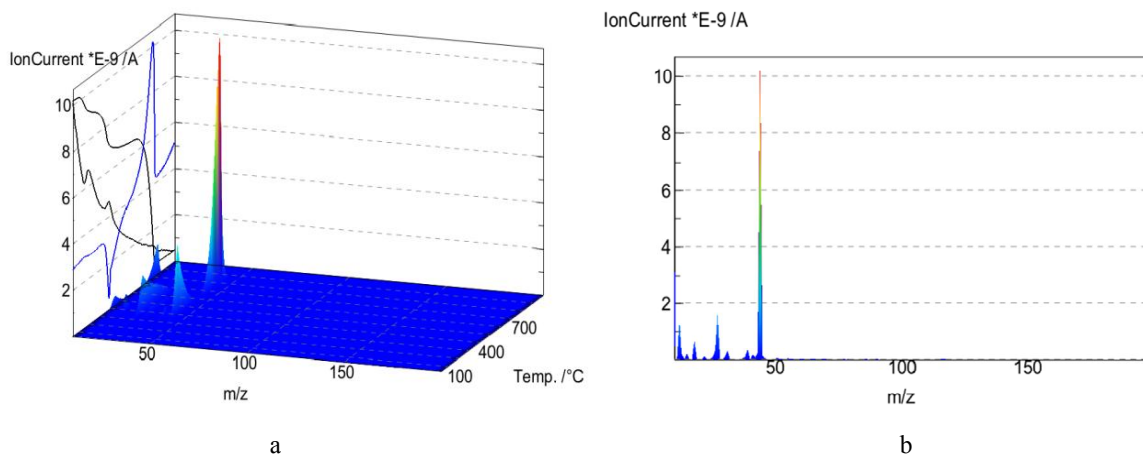


Fig. 3. TG-MS-3D (a) and MS-2D (b) of the synthesized composite

As can be seen from Fig. 3b, there are 4 peaks in the mass spectrum of the composite, which indicate that the thermal destruction of the adsorbed polymer occurs with the formation of the following compounds: the peak with mass 18 corresponds to the formation of water, the peak with mass 28 can be attributed to the formation of nitrogen N_2 and carbon monoxide CO , the peak with mass 40 can be attributed to the formation of propyne, the peak of the highest intensity with mass 44 corresponds to the release of carbon dioxide.

The values of the specific surface area of the synthesized composite, which are calculated by data processing of isotherms of nitrogen low-temperature adsorption-desorption by different methods, are shown in Table 1. The average volume and pore size of the composite surface, which are calculated from the pore distribution diagrams, are also shown in Table 1.

As can be seen from Table 1, the specific surface area of silica gel after adsorption of poly[8-oxyquinoline methacrylate] is significantly reduced (20 times), which is a logical result of fixing of a large amount of non-porous polymer on the surface. At the same time, the average pore volume decreases by about 10 times, and the average pore diameter increases by more than 2 times, which also indicates a decrease in the porosity of the silica gel surface.

Changes in the surface morphology of silica gel after adsorption of poly[8-oxyquinoline methacrylate] were examined by scanning electron microscopy (SEM).

SEM photos of the surface of the modified silica gel at an increment of 250 and 25,000 are presented in Figs. 4a and 4b, respectively. The photos show that the polymer covers most of the surface of the silica gel particles and is on the surface of the silica gel in the form of agglomerates of different geometric shapes.

Thus, the results of nitrogen low-temperature adsorption-desorption and scanning electron microscopy correlate well with each other.

Since we found²³ that *in situ* modification of silica gel surface with poly[8-oxyquinoline methacrylate] led to an improvement in its sorption properties for $Cu(II)$, $Cd(II)$, and $Fe(III)$ ions and did not improve them for Pb ions (II), it was interesting to investigate the sorption properties of silica gel with adsorbed poly[8-oxyquinoline methacrylate] relative to these ions under similar sorption conditions. Therefore, the study of the sorption properties of the synthesized composite against $Cu(II)$, $Cd(II)$, $Pb(II)$, and $Fe(III)$ ions was performed in an aqueous medium without adding any buffer to the initial solutions of nitrates of these metals for one hour with periodic mechanical shaking.

Studies have shown that the synthesized composite is characterized by a fairly high degree of sorption in the range of milligram initial concentrations of the studied ions: for Cu^{2+} ions in the range of 4–40 mg/L it is 90–96 %, for Pb^{2+} ions in the range of 3.8–38 mg/L – 71–89 %, for Fe^{3+} ions 4–40 mg/L – 91–96 % and for Cd^{2+} ions 3.8–18.5 mg/L – 93–98 %.

Table 1. Comparative characteristics of the surface parameters of the synthesized composite and the original silica gel

Sample	Surface area, m^2/g			Average pore volume cm^3/g (BJH)	Average pore size, nm (BJH)
	By isotherm	BET	Langmuir		
Original silica gel ²³	447	463	687	0.686	5.9
Synthesized composite	21	22	34	0.072	12.9

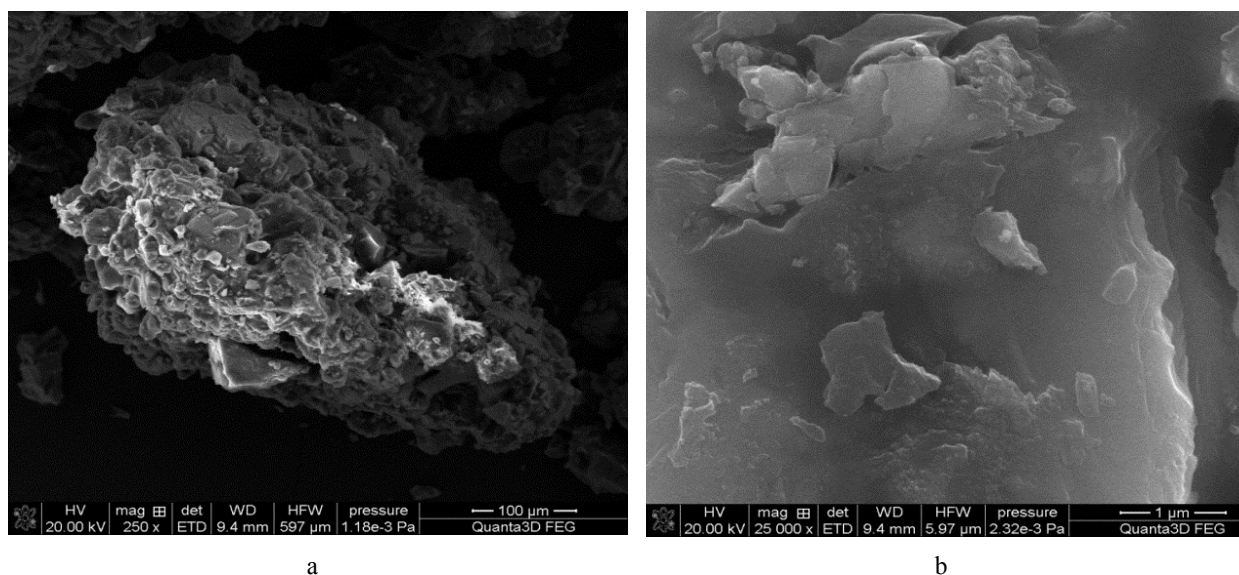


Fig. 4. SEM image of the silica gel surface after adsorption of poly[8-oxyquinoline methacrylate]:
 a – increment 250× b – increment 25000×

A sorption isotherm was obtained for each investigated metal ion. An example of such an isotherm for Cd(II) ions is shown in Fig. 5. As can be seen from Fig. 5, the obtained isotherms belong to type II according to the *IUPAC* classification. All obtained isotherms belong to type II. Based on these isotherms, the values of sorption capacity for all studied ions were found, which are shown in Table 2.

As can be seen from Table 2, the sorption capacity of silica gel after adsorption on its surface of poly[8-hydroxyquinoline methacrylate] increases for Cu(II) ions by 19.3 times compared to that for the original silica gel, relative to Pb(II) ions increases by 16.5 times, for Fe(III) ions – 15.7 times, for Cd(II) ions – 6.5 times. Comparison of the obtained data with the sorption capacity of silica gel *in situ* modified with poly[8-hydroxyquinoline methacrylate] shows that the sorption capacity of the synthesized material is 12.9 times higher than Cu(II) ions, 16.5 times higher than Pb(II) ions, for Fe(III) ions – 10.5, for Cd(II) ions – 4.9 times.

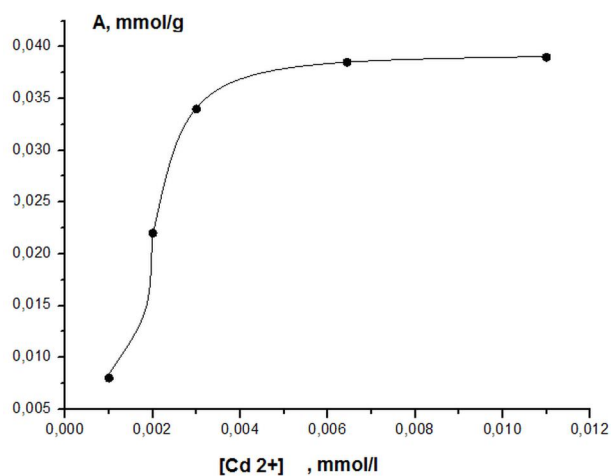


Fig. 5. Isotherm of sorption of cadmium (II) ions on the surface of silica gel with adsorbed poly[8-oxyquinoline methacrylate].
 Experimental conditions: weight of the composite – 0.1 g, sorption time – 1 hour, the volume of working solution – 50 mL

Table 2. Adsorption capacities for Cu(II), Cd(II), Pb(II), and Fe(III) ions of the synthesized composite, the original silica gel, and silica gel *in situ* modified by poly[8-oxyquinoline methacrylate]

Ion	Sorption capacity, mmol/g		
	Original silica gel	Silica gel <i>in situ</i> modified by poly[8-oxyquinoline methacrylate]	Silica gel with adsorbed poly[8-oxyquinoline methacrylate]
Cu(II)	0.006 ²³	0.009 ²³	0.116
Cd(II)	0.006 ²³	0.008 ²³	0.039
Pb(II)	0.002 ²³	0.002 ²³	0.033
Fe(III)	0.008 ²³	0.012 ²³	0.126

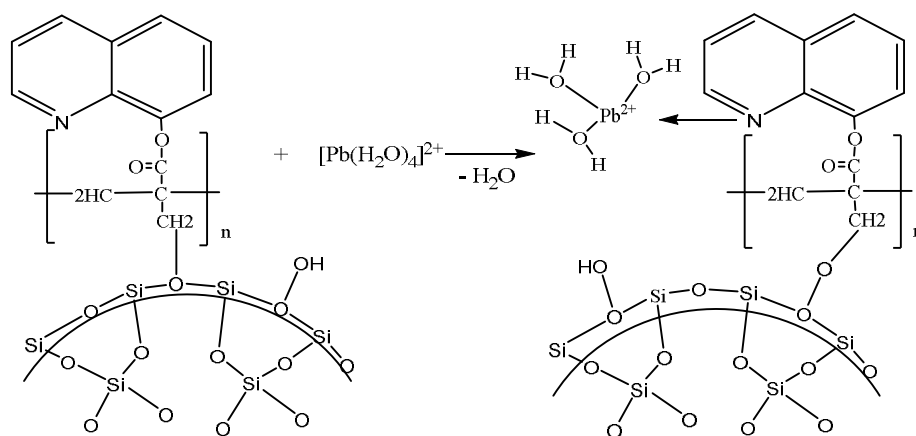


Fig. 6. Scheme of lead(II) ions sorption on the surface of silica gel with adsorbed poly[8-oxyquinoline methacrylate]

The composition of poly[8-oxyquinoline methacrylate] adsorbed on the surface of silica gel includes oxyquinoline groups, which are widely known for their complex-forming activity against the studied ions. The sorption of selected metal ions on the surface of the synthesized composite should occur mainly due to the processes of complexation of the active centers of the adsorbed polymer with metal ions. The mechanism of Cu^{2+} , Pb^{2+} , Cd^{2+} and Fe^{3+} ions sorption by the surface of silica gel with adsorbed poly[8-oxyquinoline methacrylate] is most likely as follows:

In the initial solutions, these metal ions are in the form of aqua complexes. When aqua complexes interact with the oxyquinoline groups of the adsorbed polymer, one molecule of water is replaced by an oxyquinoline group with the formation of multiligand complexes on the solid surface.

For example, for lead(II) ions, this process can be represented as shown in Fig. 6.

4. Conclusions

A new organo-mineral composite was obtained by adsorption of the synthesized poly[8-oxyquinoline methacrylate] on the silica gel surface.

Immobilization of poly[8-oxyquinoline methacrylate] on the silica surface was confirmed using IR spectroscopy and thermogravimetric analysis combined with mass spectrometry. According to thermogravimetric analysis, it was found that the mass fraction of adsorbed poly[8-oxyquinoline methacrylate] in the composite is 43.5 %.

From the analysis of photographs of the synthesized composite surface obtained by scanning electron microscopy, it follows that poly[8-oxyquinoline methacrylate] covers the majority of the surface of silica gel

particles. The polymer is located on the surface of silica gel in the form of agglomerates of different geometric shapes.

It was found that the sorption capacity of the synthesized material in comparison with the capacity for the original silica gel is significantly higher for all studied ions and higher than for silica gel *in situ* modified with poly[8-oxyquinoline methacrylate], for Cu (II) ions – 12.9 times, for Pb (II) ions – 16.5, for Fe (III) ions – 10.5, for Cd (II) ions – 4.9 times.

The prepared sorbent can be considered a promising sorbent for the selective separation of Cu (II), Cd (II), Pb (II), and Fe (III) from wastewater samples.

Data availability

The authors declare that the data supporting the findings of this study are available within the article.

Conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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СИНТЕЗ, СОРБЦІЙНІ ВЛАСТИВОСТІ ТА ОЦІНКА СИЛКАГЕЛЮ З АДСОРБОВАНИМ ПОЛІ[8-ОКСИХІНОЛІНМЕТАКРИЛАТОМ] ЯК СОРБЕНТУ ІОНІВ Cu(II), Cd(II), Pb(II) ТА Fe(III)

Анотація. У цьому дослідженні новий функціоналізований кремнеземний композит отриманий у результаті адсорбції полі[8-оксихінолінметакрилату] на поверхні силкагелю. Як адсорбент для видалення з води іонів Cu(II), Cd(II), Pb(II), Fe(III) ми використовували полімерно функціоналізований силкагель. Імобілізація полі[8-оксихінолінметакрилату] на поверхні силкагелю була підтверджена за допомогою ІЧ-спектроскопії та термогравіметричного аналізу в поєднанні з мас-спектрометрією. Скануюча електронна мікроскопія показала, що полімер майже повністю покриває поверхню силкагелю. В результаті значно зменшується пористість поверхні мінералу. Досліджено процеси сорбції іонів Cu(II), Cd(II), Pb(II), Fe(III) на поверхні синтезованого композиту в статичному режимі.

Ключові слова: адсорбція, композит, силкагель, полі[8-оксихінолінметакрилат], іони Cu(II), Cd(II), Pb(II), Fe(III).